



LAWRENCE
LIVERMORE
NATIONAL
LABORATORY

BDS Thin Film UV Antireflection Laser Damage Competition

C. J. Stolz

November 4, 2010

SPIE Laser Damage Conference
Boulder, CO, United States
September 26, 2010 through September 29, 2010

Disclaimer

This document was prepared as an account of work sponsored by an agency of the United States government. Neither the United States government nor Lawrence Livermore National Security, LLC, nor any of their employees makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States government or Lawrence Livermore National Security, LLC. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States government or Lawrence Livermore National Security, LLC, and shall not be used for advertising or product endorsement purposes.

BDS Thin Film UV Antireflection Laser Damage Competition

Christopher J. Stolz
Lawrence Livermore National Laboratory,
7000 East Avenue, L-491, Livermore, CA 94550

Mark Caputo, Andrew J. Griffin, and Michael D. Thomas
Spica Technologies, Inc.
18 Clinton Drive, Suite #3, Hollis, NH 03049

ABSTRACT

UV antireflection coatings are a challenging coating for high power laser applications as exemplified by the use of uncoated Brewster's windows in laser cavities. In order to understand the current laser resistance of UV AR coatings in the industrial and university sectors, a double blind laser damage competition was performed. The coatings have a maximum reflectance of 0.5% at 355 nm at normal incidence. Damage testing will be performed using the raster scan method with a 7.5 ns pulse length on a single testing facility to facilitate direct comparisons. In addition to the laser resistance results, details of deposition processes and coating materials will also be shared.

Keywords: laser damage testing, antireflection coating, thin film, multilayer, ultraviolet, nanosecond laser

1. INTRODUCTION

Over the last three years, a thin film laser damage competition has been held at the Boulder Damage Symposium in an attempt to gain a better understanding of the current state of high laser resistant thin films. Additionally the purpose of this series of competitions is to determine general trends regarding deposition methods and coating materials over a range of pulse lengths and wavelengths. The first competition was a 1064 nm high reflector tested with a 5 ns pulse length laser.¹ The second competition was also a high reflector, however, given the increased interest in femtosecond lasers, a 786 nm high reflector was damage tested with a 200 femtosecond pulse length laser.² This year, antireflection coatings were tested at 355 nm with a 7.5 ns pulse length since little work has been done in this area over the last few decades.³⁻⁵ In each case, the damage threshold distribution range was spread at least an order of magnitude indicating a widespread difference in process knowledge for generating high laser resistant coatings.

2. PARTICIPATION

Twenty-nine samples were submitted to this competition from eleven different companies or institutes listed in table 1. Both uncoated and antireflection coated samples were submitted by each participant in an attempt to deconvolute the potential substrate influence on the coating laser damage threshold results. The participants came from five different countries; USA (13), Germany (5), China (5), Japan (4), and the United Kingdom (2) representing North America, Europe, and Asia.

Table 1 List of participating companies or institutes for the BDS thin film damage competition.

Absolute Coatings	Gooch & Housego, General Optics	Layertec Optical
Arrow Thin Films	Jiutle	Nikon Corporation
Gooch & Housego	Laser Zentrum Hanover	Optical Coatings Japan
Gooch & Housego, Cleveland Crystals	Lawrence Livermore National Laboratory	

3. SAMPLES

Unlike the high reflector mirror case where a very low electric field occurs at the substrate film interface for a non-defective coating, high electric fields occur at the substrate film interface in antireflection coatings. Therefore, the substrate can have a significant impact on the laser resistance of an antireflection coating. For this competition the participants were asked to contribute both coated and uncoated samples with the intention of increasing understanding of the impact of the AR coating on the laser damage resistance of the coated optic.

The spectral requirement was a reflection less than 0.5% at 351 nm at normal incidence. Environmental requirements were ambient lab conditions (40% relative humidity and a temperature of 20 degrees Celsius). There were no stress or reflected wavefront requirements. Substrates were supplied by the participant with dimensions of 50 mm in diameter and 10 mm thick. The substrate material was fused silica. Participants were asked to provide a description of the deposition process, a list of the coating materials, a plot to validate spectral performance, and the layer count.

Samples were removed from participant supplied packaging containers into identical PETG packaging containers in an attempt to remove any identification link to the supplier. Also, for anonymity, a unique code was assigned to each sample. The identity of the suppliers and sample was kept by an administrative assistant to maintain a double blind experiment. The author and damage testing service did not have access to the identity of any of the samples so as to remain unbiased and to protect the identities of participants whose samples were the least laser resistant.

Six different coating deposition techniques were used to manufacture the submitted samples as shown in figure 1. The samples were deposited by electron beam deposition, ion beam sputtering, magnetron sputtering, resistive evaporation, or sol gel (dip or spin). Some of the e-beam coatings were densified by ion assistance.

Six different coating materials were used to manufacture the samples as illustrated in figure 2. Silica (SiO_2) was used in most of the samples through either physical vapor deposition or porous silica for the sol gel coatings. The other low index materials that were used were fluorides, which are known for their transmission into the deep UV, including magnesium fluoride (MgF_2) and lanthanum fluoride (LaF_3). The two high index materials were hafnia (HfO_2) and scandia (Sc_2O_3). Alumina (Al_2O_3) is the final material that was used in two of the samples. It is a medium refractive index material with a transmission deep into the UV. One participant declined to provide the coating materials used in their contribution.

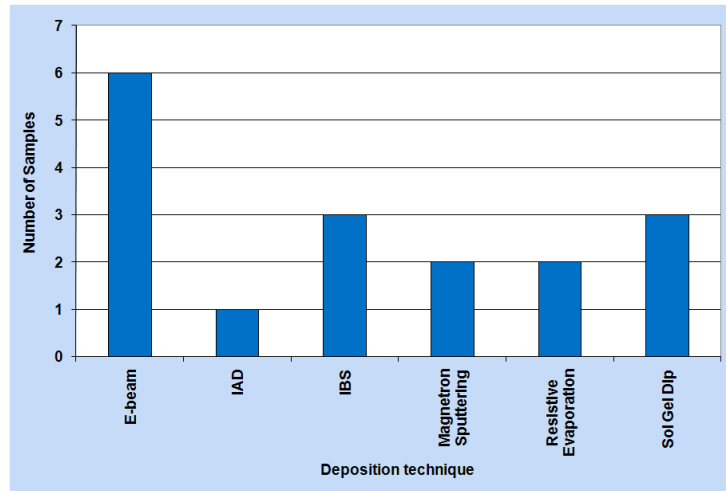


Fig. 1 Distribution of deposition technologies for the contributed samples.

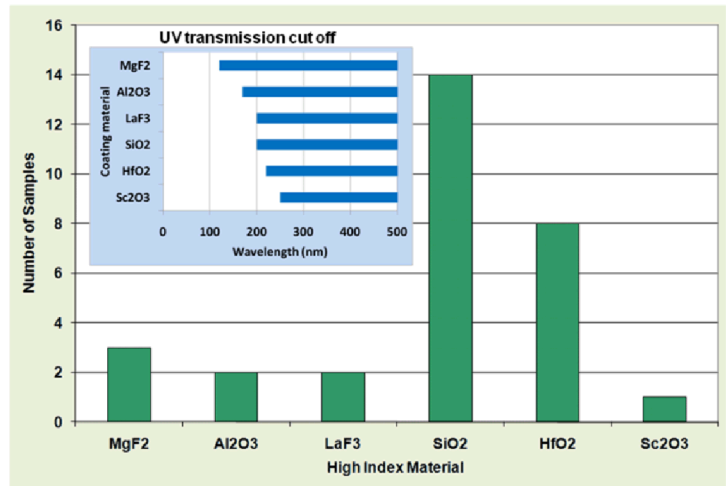


Fig. 2 Distribution of high index materials for the contributed samples.

4. DAMAGE TESTING

The samples were damage tested at Spica Technologies according to the method described by Borden.⁶ The pulse length of the testing laser was 7.5 ns with a repetition rate of 10 Hz. The wavelength was 355 nm. The beam diameter was nominally 0.58 mm at $1/e^2$. The samples were raster scanned over a 10 mm by 10 mm area starting at 1 J/cm² and increasing in 1 J/cm² increments up to 6 J/cm². Beyond 6 J/cm² the increment was increased to 2 J/cm² to minimize the testing time. For some of the uncoated samples an increment of 5 J/cm² was used at higher fluences. The beam was translated by the beam diameter at the 90% intensity to achieve a uniform intensity across the test area. Laser damage was detected by scatter of a HeNe laser probe beam observed with a microscope imaged at the sample plane with a CCD.

Damage was classified into three categories, “No Damage”, “Initiation”, and “Failed”. “No Damage” is defined as no visible change to the coating. “Initiation” is defined as the observation of pinpoints as large as 100 μm , however, none of the pinpoint damage grew upon repeated illumination. “Fail” is defined as the fluence where pinpoint damage exceeded 100 μm , pinpoint damage grew upon repeated illumination, or pinpoint damage occurred in more than 1% of the total number of sites.

5. Results

A wide range in laser damage threshold was observed for the coated and uncoated samples as can be observed in figure 4. This wide range implies that there are significant differences within the coating industry in the understanding of the critical process parameters necessary to manufacture high laser resistant UV antireflection coatings. An additional striking observation from figure 4 is the consistent high laser resistance of sol gel coatings. Although these coatings are mechanically weak and prone to spectral degradation in the presence of outgassing contaminants, they remain the deposition process of choice for most large high energy laser systems across the world. Sol gel coatings have the advantage of being single layer coatings due to the extremely low refractive index achieved by a porous silica

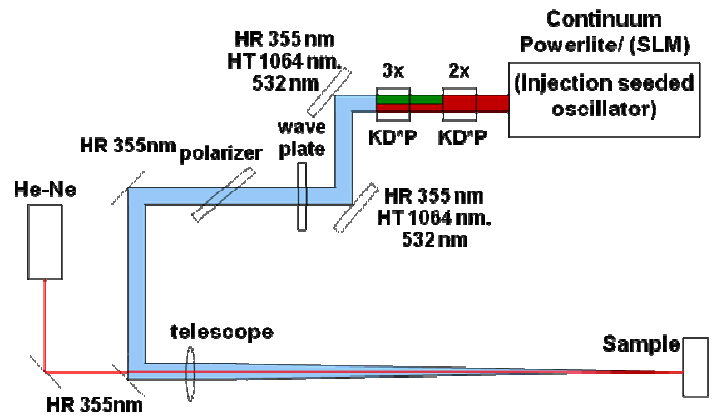


Fig. 3 Schematic of the LIDT measurement set-up.

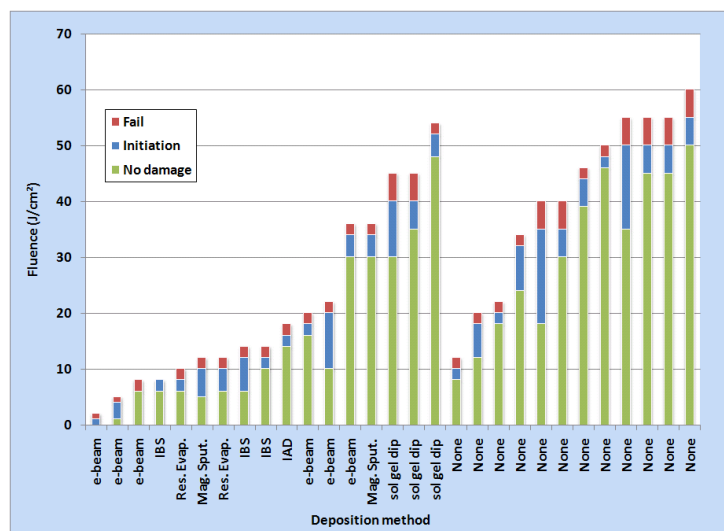


Fig. 4 Distribution of laser resistance as a function of deposition process.

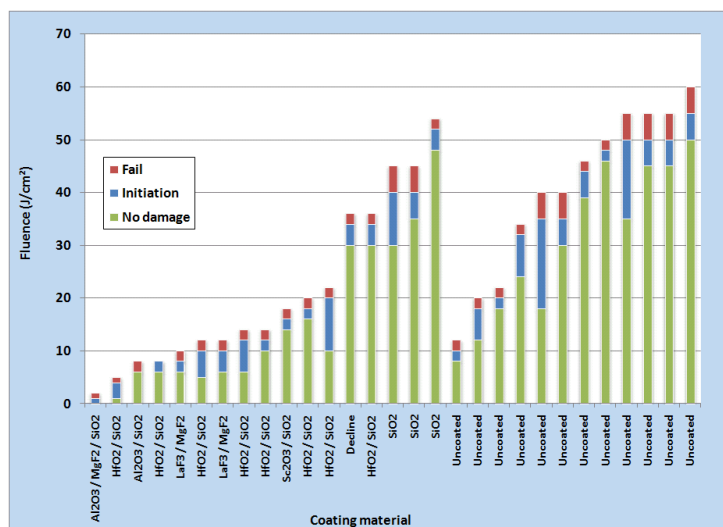


Fig. 5 Distribution of laser resistance as a function of coating material.

layer, eliminating the need for the low laser resistant high or medium refractive index materials.

Another result of the data plotted in figure 4 is the lack of significant difference between the best magnetron sputtered and electron beam deposition coatings suggesting that it is the process details that are more important than the process type for this class of coating. Only two different participants contributed coatings deposited by ion beam sputtering and one participant submitted coatings deposited by resistive evaporation. Perhaps more favorable results would be seen with these two different deposition techniques if more participants utilizing these technologies would have participated.

The antireflection coatings with silica tended to have the best laser resistance as illustrated in figure 5. Multilayer coating designs incorporating hafnia tended to have the highest laser resistance. Surprisingly the fluoride and alumina coatings did not perform better in spite of their high UV transmission and significant development of fluorides for UV mirrors.⁴ Coatings with scandia also tended to be less laser resistant, but the limited sample number makes it difficult to draw any meaningful conclusions about the effectiveness of this material.

Simpler coatings tended to be the most laser resistant as illustrated in figure 6. However, the trend of laser damage resistance versus number of coating layers is not very strong.

A much stronger damage threshold trend is observed in figure 7 which plots the difference in the laser damage threshold between the best coated sample submitted by each participant and the laser damage threshold of their uncoated sample. The majority of the coated samples in this competition had lower thresholds than their sister uncoated sample. A negative value indicates that the antireflection coating had a lower laser resistance than the uncoated sample which tended to be the case for most of the samples. Obviously a large negative value indicated a very high laser resistance of the uncoated sample and very low laser resistance of the coated sample. Certainly some of the participants have excellent polishing technology, but have not invested similar efforts into their UV antireflection coating technology. A small magnitude change (particularly a negative value) indicates that the coatings are either well matched to the substrates or possibly that the coating laser resistance could be improved with better quality substrates. A positive difference indicates an inconsistent quality between substrates, or the more unlikely conclusion that the coating somehow increases the laser resistance of the surface. Figure 8 also illustrates the difference between the laser resistance of the coated and uncoated samples.

Additionally figure 8 illustrates the impact of minor process changes on UV antireflection coating laser resistance. The use of a mixture for the high index material for participant A yields a more laser resistant coating for an IBS

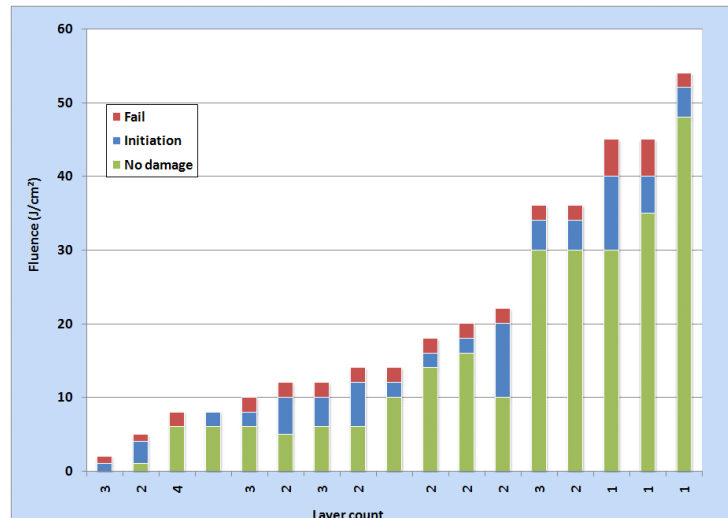


Fig. 6 Fluence distribution as a function of layer count suggest that AR coatings with fewer layers tend to be more laser resistant.

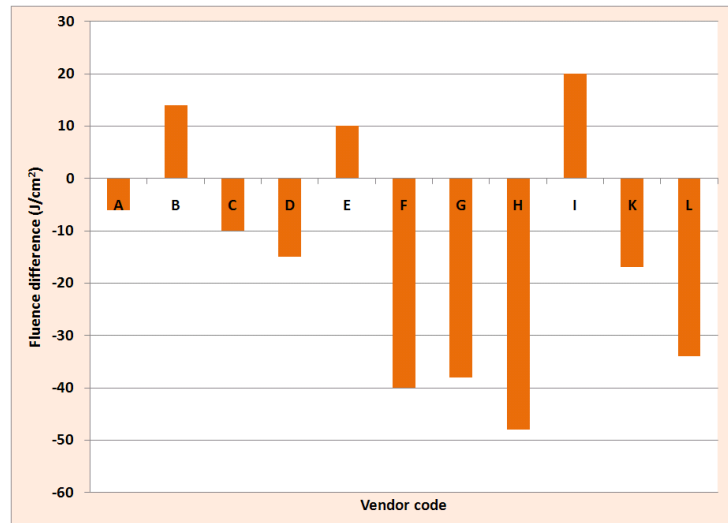


Fig. 7 Fluence difference between uncoated and coated samples by contributing participant. A large negative difference indicates a significant reduction in the coating damage threshold over the uncoated sample.

process. Participant I explored the impact of different process temperatures for resistive deposition of fluorides and observed minimal changes.

6. CONCLUSIONS

Ultraviolet antireflection coatings damage tested with nanosecond pulses have a wide range of laser resistance from a number of participants. Sol gel coatings currently have the highest laser resistance of any of the deposition processes submitted. Silica and Hafnia coating materials tended to yield the highest laser resistance compared to the fluorides. There was not a correlation between the UV transmission cutoff and the laser resistance for the different coating materials. Similar laser resistance between two radically different processes (IBS and e-beam) suggest that process parameters, such as substrate preparation, particle generation, or material stoichiometry may be playing a significantly greater role in laser resistance of antireflection coatings than the deposition technique.

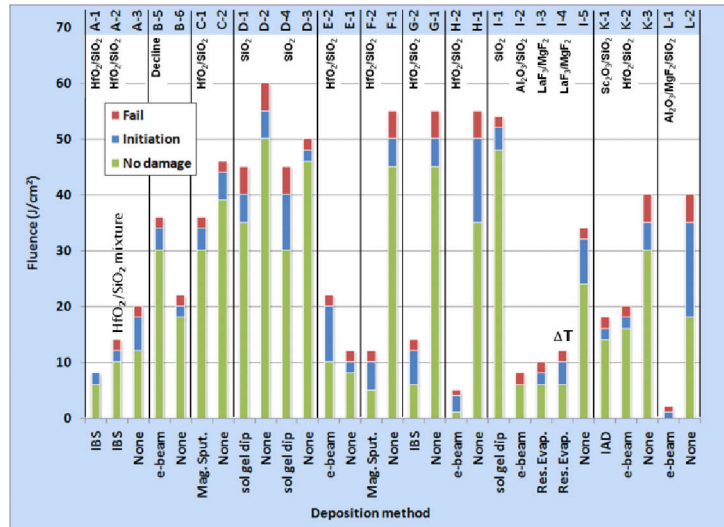


Fig. 8 Direct comparison of coating process parameters by participant.

ACKNOWLEDGEMENTS

The authors would like to acknowledge all of the participants who prepared the samples and provided the information about how the coatings were manufactured. These samples represent a significant investment to manufacture by the companies that participated. Spica Technologies Inc. graciously donated all of the laser damage testing which is also a significant investment. Finally, Artika Lal organized the data and samples to maintain a double blind experiment.

REFERENCES

1. C. J. Stolz, M. D. Thomas, and A. J. Griffin, "BDS thin film damage competition," in *Laser-Induced Damage in Optical Materials: 2008*, G. J. Exarhos, D. Ristau, M. J. Soileau, and C. J. Stolz, eds., Proc. SPIE 7132, 71320C-1-6 (2009).
2. C. J. Stolz, D. Ristau, M. Turnowski, and H. Blaschke, "Thin film femtosecond laser damage competition," in *Laser-Induced Damage in Optical Materials: 2009*, G. J. Exarhos, V. Gruzdev, D. Ristau, M. J. Soileau, and C. J. Stolz, eds., Proc. SPIE 7504, 75040S-1-6 (2010).
3. F. Rainer, L. J. Atherton, J. H. Campbell, F. P. De Marco, M. R. Kozlowski, A. J. Morgan, and M. C. Staggs, "Four harmonic database of laser-damage testing," in *Laser-Induced Damage in Optical Materials: 1991*, H. E. Bennett, L. L. Chase, A. H. Guenther, B. E. Newnam, and M. J. Soileau, eds., Proc. SPIE 1624, 116-127 (1992).
4. F. Rainer, F. P. De Marco, M. C. Staggs, M. R. Kozlowski, L. J. Atherton, and L. M. Sheehan, "A historical perspective on fifteen years of laser damage thresholds at LLNL," in *Laser-Induced Damage in Optical Materials: 1993*, H. E. Bennett, L. L. Chase, A. H. Guenther, B. E. Newnam, and M. J. Soileau, eds., Proc. SPIE 2114, 1-22 (1994).
5. H. Schink, J. Kolbe, F. Zimmermann, D. Ristau, and H. Welling, "Reactive ion beam sputtering of fluoride coatings for the UV/VUV range," in *Laser-Induced Damage in Optical Materials: 1990*, H. E. Bennett, L. L. Chase, A. H. Guenther, B. E. Newnam, and M. J. Soileau, eds., Proc. SPIE 1441, 327-338 (1991).
6. M. R. Borden, J. A. Folta, C. J. Stolz, J. R. Taylor, J. E. Wolfe, A. J. Griffin, and M. D. Thomas, "Improved method for laser damage testing coated optics," in *Laser-Induced Damage in Optical Materials: 2005*, G. J. Exarhos, A. H. Guenther, K. L. Lewis, D. Ristau, M. J. Soileau, and C. J. Stolz, eds., Proc. SPIE 5991, 59912A-1-8 (2006).